

4. COMPARISONS OF KEY RESULTS

Samples from the ICET tests were subjected to numerous laboratory analyses. This section presents key results from these analyses and compares them for each test. Section 4.1 presents water chemistry and precipitate analyses, Section 4.2 discusses the findings from examinations of fiberglass and cal-sil samples, Section 4.3 describes the post-test sediment, Section 4.4 compares photographs and microscopic analyses of coupons, Section 4.5 discusses results from deposition product examinations, and Section 4.6 presents analyses of the Test #3 gel-like precipitate.

4.1. Water Samples

Daily water samples were obtained during each ICET test. Numerous water chemistry parameters were measured and are presented in Section 4.1.1. Additionally, precipitates were observed in Tests #1 and #5 and are discussed in Section 4.1.2.

4.1.1 Water Chemistry

Throughout the ICET test series, daily measurements of pH, turbidity, TSS, and kinematic viscosity were taken. Additionally, daily measurements of selected elemental concentrations were obtained. In this section, these measurements are compared. Since chemical parameters and insulation debris differed from test to test, Table 4-1 was compiled to serve as a guide to system parameters as the water chemistry results are discussed.

Table 4-1. Test Conditions in the ICET Tests.^{1,2}

Test #	Boron ³ added (mg/L)	NaOH added (mg/L)	TSP ⁴ added (mg/L)	Controlling pH buffer system	Insulation ⁵	
					Fiberglass	Cal-sil
1	2,800	7,677	-	borate	100%	-
2	2,800	-	4,000	phosphate	100%	-
3	2,800	-	4,000	phosphate	20%	80%
4	2,800	9,600	-	borate	20%	80%
5	2,400	-	-	borate	100%	-

¹The chemical doses were according to the required parameters given in Table 2-4. To determine the quantities of NaOH and TSP required to meet the target pH values in Table 2-4, water quality modeling using Visual Minteq Version 2.30 was conducted, followed by verification through bench-scale testing prior to the ICET tests.

²Temperature was 60°C ± 3°C in all tests. The following chemicals were also added to the solution: LiOH = 0.7 mg/L and HCl = 100 mg/L in Tests #1–#4. LiOH = 0.3 mg/L and HCl = 42.8 mg/L in Test #5. Also, 63.7 g of latent debris and 21.21 g concrete dust was added to the solution prior to the test initiation for each test.

³The required concentration of boron was added as 16,000 mg/L H₃BO₃ in Tests #1–#4, and as 6,848 mg/L H₃BO₃ and 10,580 mg/L as Na₂B₄O₇·10H₂O in Test #5.

⁴TSP = Na₃PO₄·12H₂O (Trisodium phosphate).

⁵All tests consisted of 0.137 cu ft of insulation represented per cubic foot of water. For each test, the insulation was present as fiberglass and/or calcium silicate material with the distribution of materials as shown in the table.

Each test had a different pH that was attributable to specific chemical requirements (Table 4-1). As shown in Figure 4-1, although the target pH was different between tests, all tests had a similar consistency with only a slight drift in pH throughout the test. The only exception to this trend was observed in Test #3, where the pH deviated from the initial pH by approximately 0.8 unit. In Test #3, phosphate precipitated out of solution early during the test which resulted in the diminished buffering capacity of the system. Without adequate buffering, the system pH could be more easily affected as varying chemical reactions occurred.

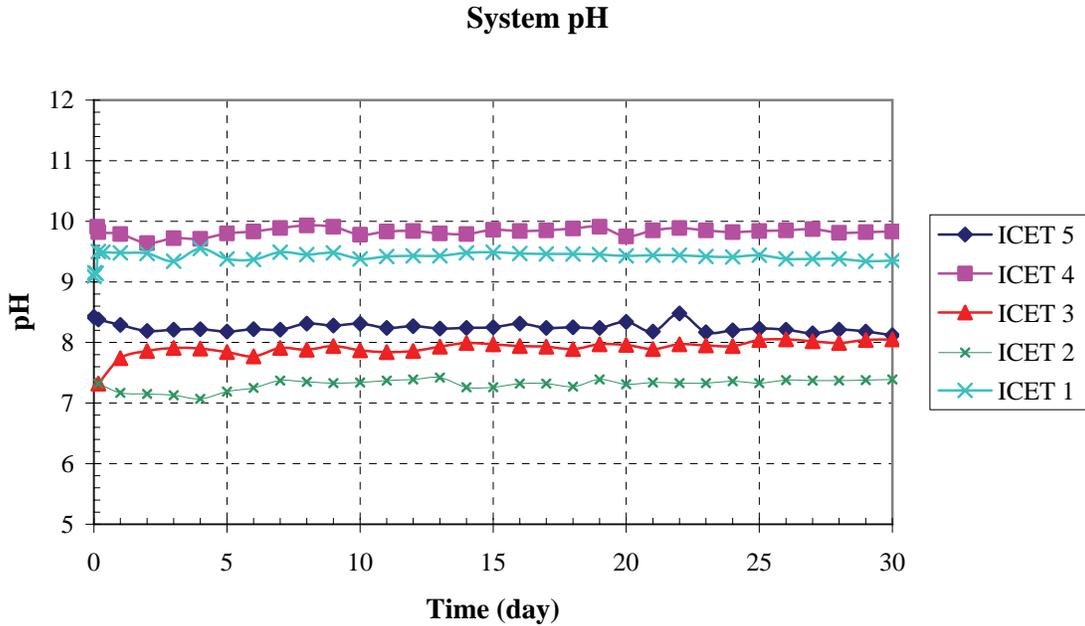


Figure 4-1. ICET bench-top pH comparison.

Turbidity measurements were taken frequently at 60°C during the first day of testing and are presented in Figure 4-2. The number of samples taken during the first day of each test varied. The official procedure only specified a time-zero, 4-hour, and 24-hour sample, but as a result of visual observations and varying test ingredients, samples were taken more often for some tests.

Day One Turbidity

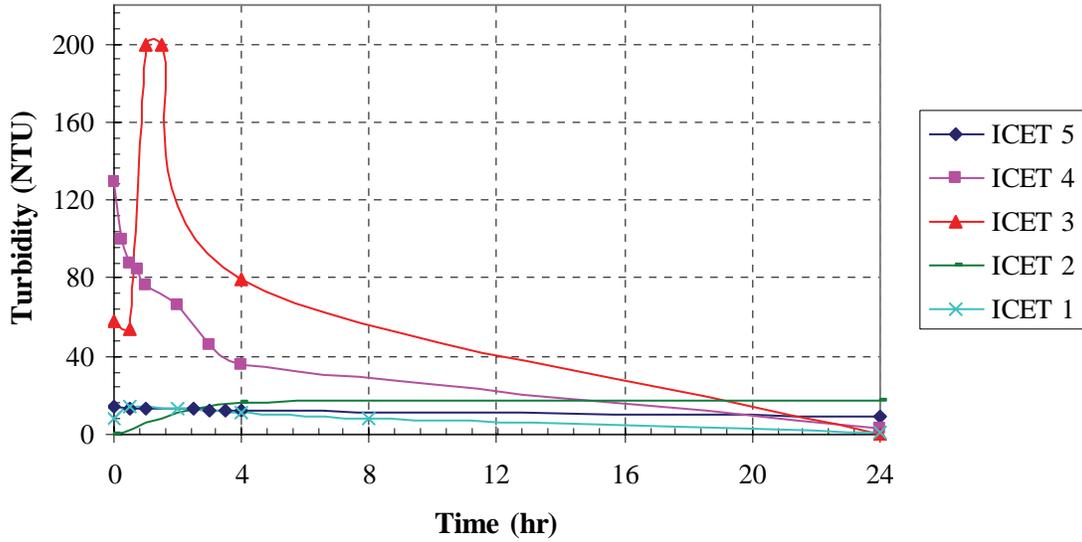


Figure 4-2. Day 1 turbidity.

In all of the tests, turbidity peaked within the first few hours of testing and decreased to lower values within 24 hours. Tests #3 and #4 had higher time zero measurements as compared with the other tests because cal-sil dust was added to the tank before test initiation. The difference in the peak measurements is because of the varying chemical and insulation requirements for each test.

For all tests, the 60°C turbidity values, (Figure 4-3), were relatively similar. Each reached a maximum value during first day of testing and by the second day of testing decreased to very low values which were similar to the baseline measurements. The turbidity measurements remained at these lower values for the duration of the tests.

60 °C Turbidity Measurements

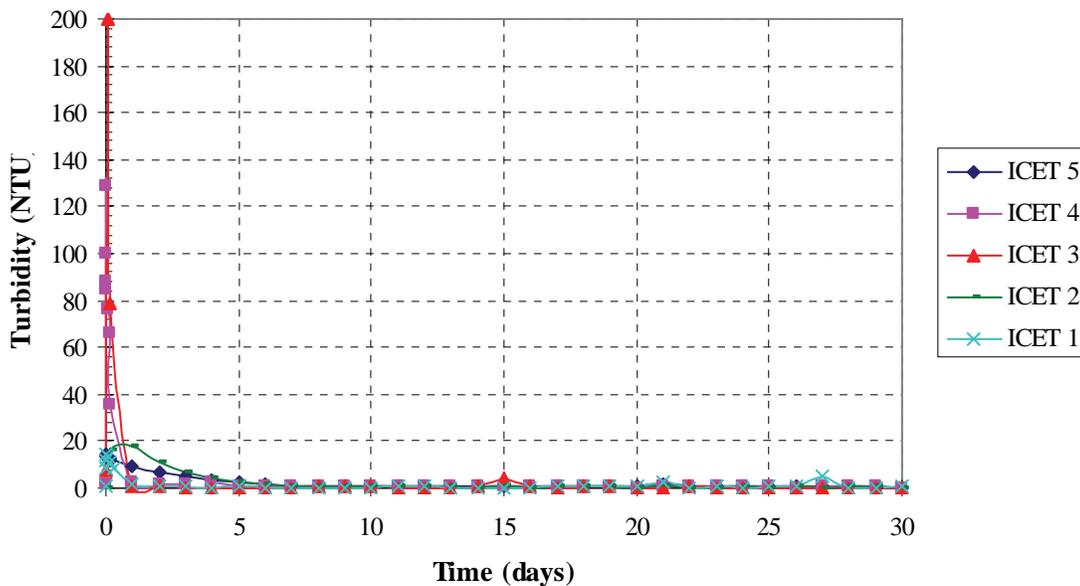


Figure 4-3. ICET 60°C turbidity.

Turbidity measurements at 23°C, as shown in Figure 4-4, were also relatively similar for Tests #2–#4. Turbidity reached a maximum value within the first day of testing and decreased towards the base-line value, with Test #2 having the longest delay time. Tests #1 and #5 turbidity measurements deviated from the previously observed pattern, which may be attributable to temperature-dependent solution behavior. Tests #1 and #5 solutions produced precipitates upon cooling that made the solutions cloudy in nature and consequently provided higher turbidity readings. Since Test #1 had a larger concentration of soluble aluminum and a larger amount of subsequent precipitation, it also experienced a more dramatic deviation in turbidity. As Test #1 progressed, the cloudy nature of the cooled solution became more pronounced, which is indicative of increased soluble aluminum concentration resulting in increased precipitation at room temperature.

23 °C Turbidity Measurements

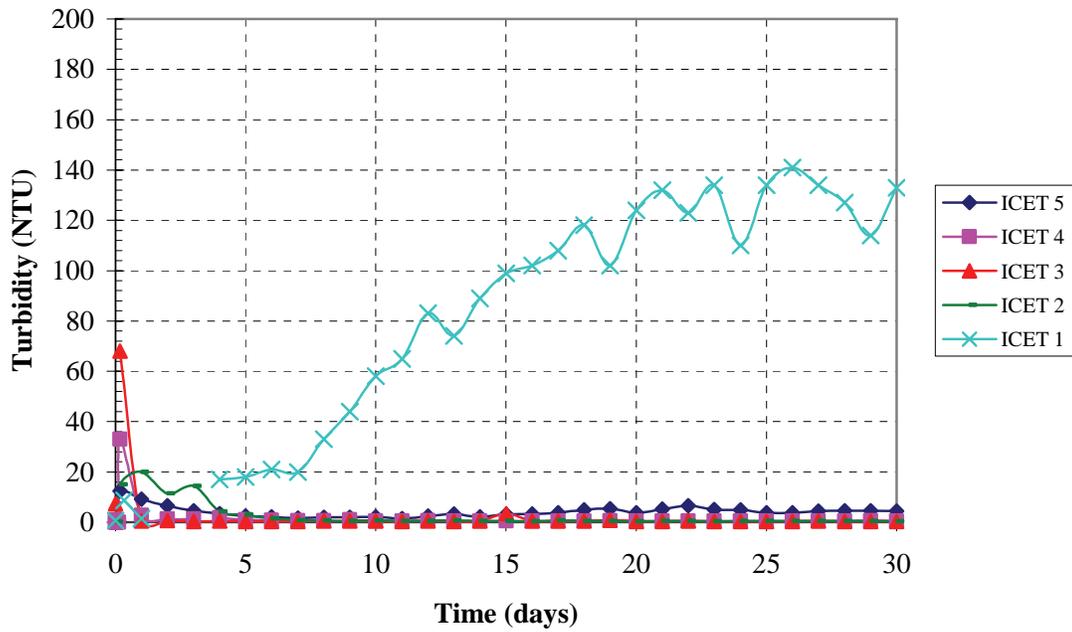


Figure 4-4. ICET 23°C turbidity.

TSS measurements were also taken for each test. As can be seen in Figure 4-5, each test reached a maximum value within the first few hours of testing and declined to a minimum value similar to the base-line measurement. As seen in Figure 4-6, the daily TSS measurements varied within a moderate range around the minimum value obtained during the first day of testing. Tests #3 and #4 provided the largest initial TSS concentrations, which are attributable to the addition of cal-sil into the chemical environment prior to test initiation.

Day One TSS

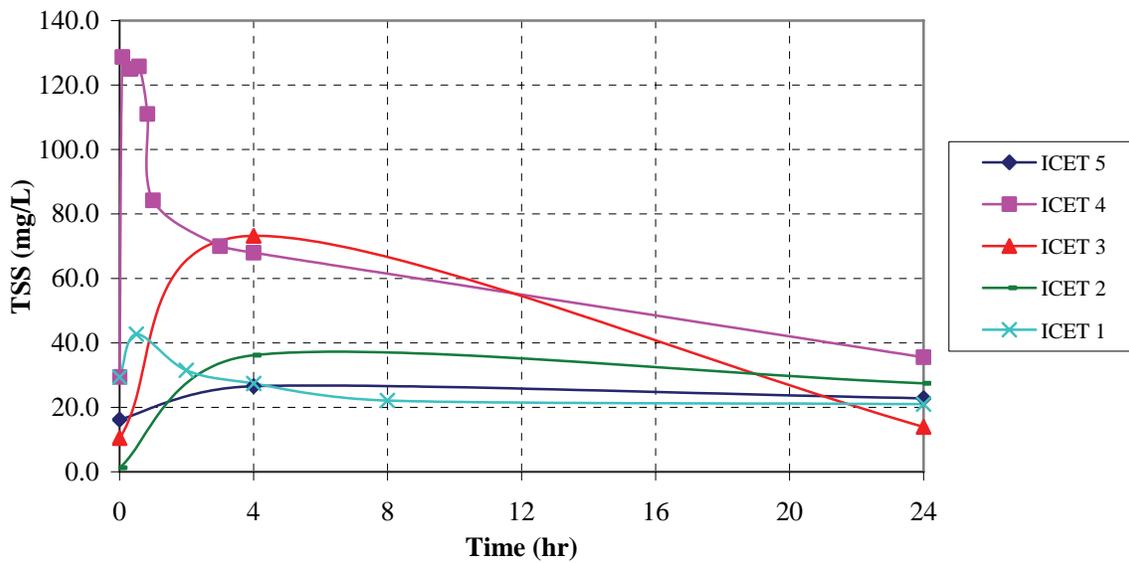


Figure 4-5. ICET Day-1 TSS.

TSS

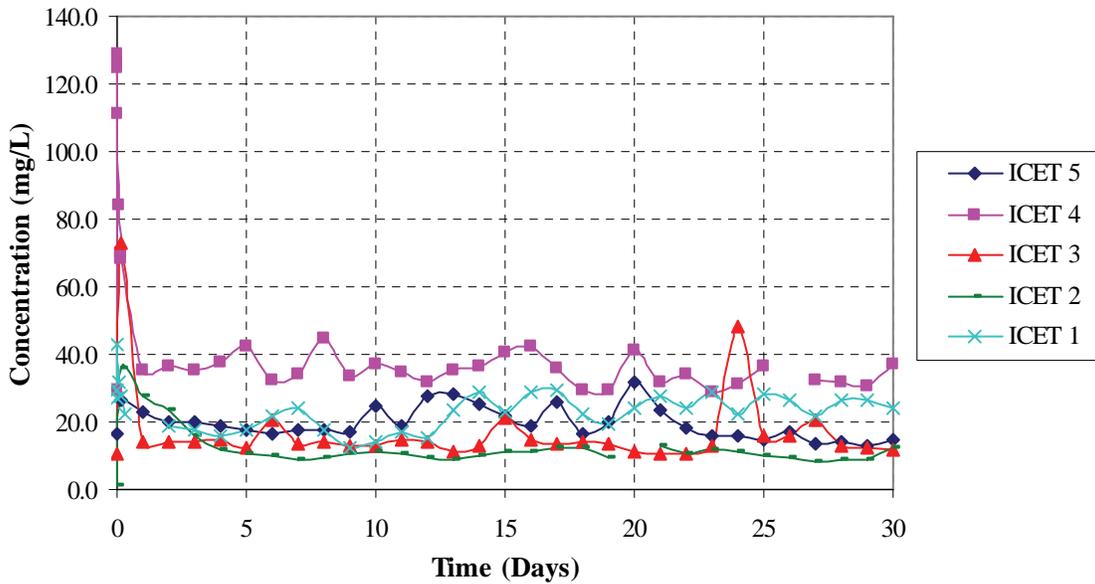


Figure 4-6. ICET TSS.

Measurements of the kinematic viscosity taken at 60°C resulted in approximately constant values, with the exception of Test #1 (Figure 4-7). All values were greater than that of pure water (0.4665 mm²/s at 60°C, see Ref. 9). The highest viscosities were obtained in Tests #1 and #4,

which may be attributed to the higher concentrations of suspend solids in solution (Figure 4-6) and/or to the large concentrations of chemicals added to solution (Table 4-1). The viscosity of the bulk solution can be affected by the presence of particulates, electrolytes, and gelatinous substances.

60 °C ICET Viscosity

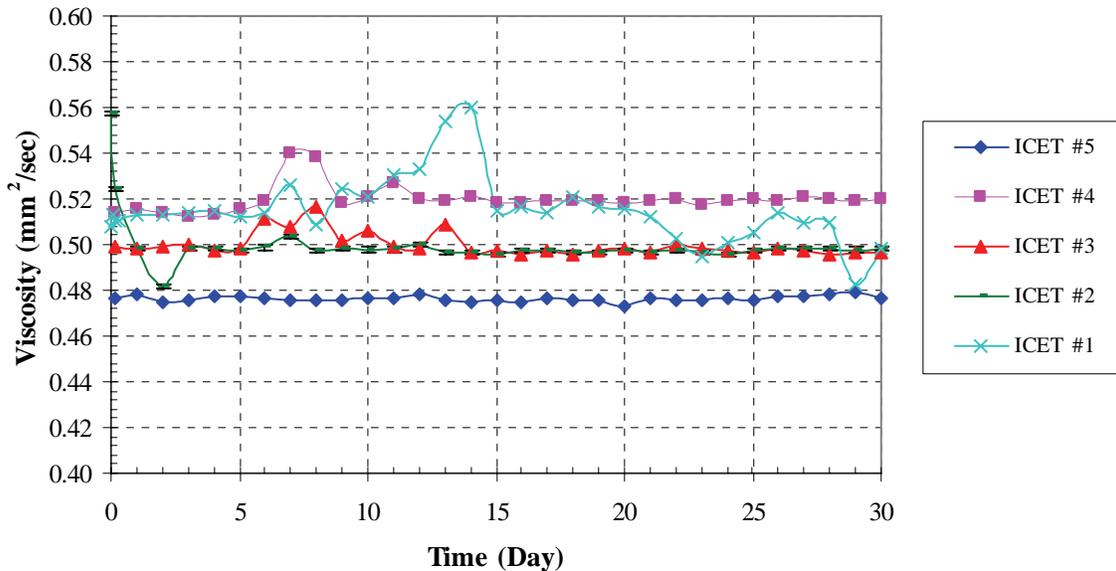


Figure 4-7. ICET 60°C kinematic viscosity.

The room-temperature kinematic viscosities (Figure 4-8) were also relatively constant with the exception of Test #1. Although the room temperature measurements taken during ICET #1 experienced deviations from day to day, the measurements generally increased to a higher value throughout the duration of the test. The increase is attributable to the increase in aluminum concentration and visible precipitates. The variations within the Test #1 viscosity measurements could be attributed to temperature-dependent solution behavior. Following Test #1, daily measurement protocols were refined to report the kinematic viscosity after a 5-minute cooling time in a constant-temperature water bath.

23 °C ICET Viscosity

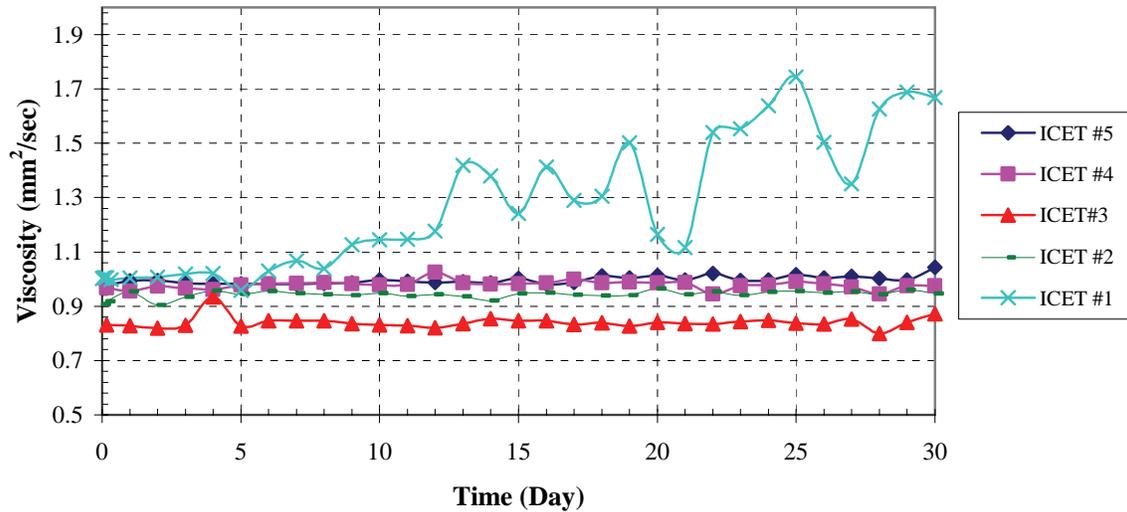


Figure 4-8. ICET 23°C kinematic viscosity.

As previously noted, the Test #1 solution became cloudy when its temperature was reduced. This cloudiness is attributable to chemical product precipitation. The technique used to measure viscosity was not intended to obtain measurements of solution with precipitation present. Consequently, for the systems in which precipitation was observed, the viscosity measurements should only be interpreted qualitatively.

During the ICET test series, the hydrogen concentration was measured in the tank atmosphere. The tank was passively vented during all tests to avoid an unsafe hydrogen buildup. However, as shown in Figure 4-9, differences were noted between tests, with Test #1 having the highest concentration. Because of changes in operational procedures, passive venting, and the simplicity of the measurement tool, these measurements should be used for qualitative interpretations only.

Hydrogen Generation

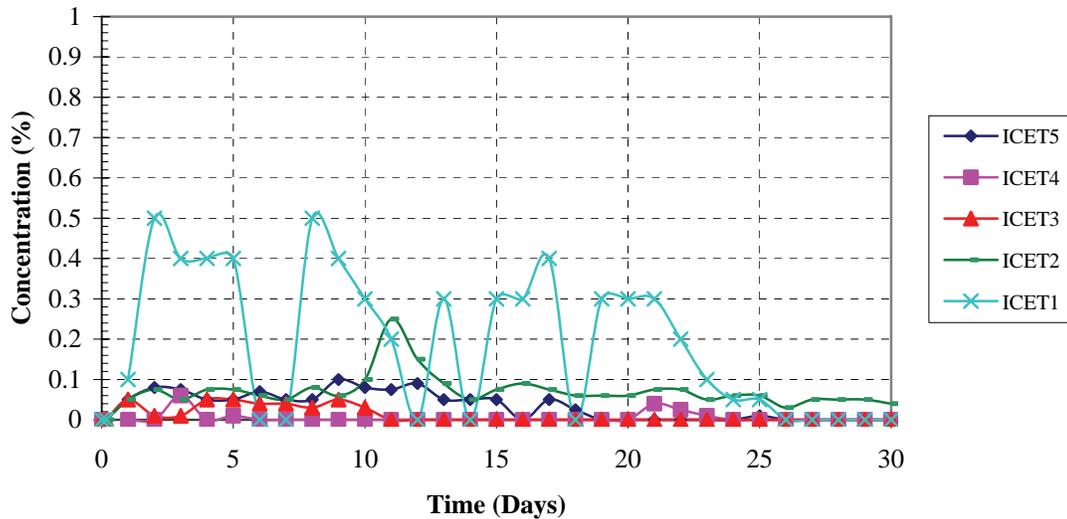


Figure 4-9. ICET hydrogen concentration.

During Test #1, ventilation valves were initially kept closed and only opened for hydrogen measurements and for venting when values reached a level approaching the safety action threshold. Within the first few days of the test, the hydrogen measurements were above the safety action threshold of 0.4%. These measurements were checked by UNM health and safety officials and found to be erroneous, which resulted in a procedural change for hydrogen sampling. An evaluation of the Test #1 hydrogen concentrations was made, keeping in mind the limitations of the measurement and the changes in operational procedures. It is speculated that the zero values on Days 6, 7, 12, 14, and 18 were erroneous. However, the trend seen from Day 20 until the end of the test is qualitatively accurate. Of interest is the hydrogen generation decreasing to zero from Day 21 to Day 26, which corresponds to the leveling off of aluminum concentration on Day 25 (Figure 4-10).

Another procedural change was made at the start of Test #2. For the first 10 days of testing, the vent valves on the tank lid were left open at all times and the hydrogen generation measurements were relatively low (0.05%–0.1%). For Day 11 through Day 12, the vent valves were closed to determine actual hydrogen build-up in the tank which spiked at 0.25%. For Tests #3–#5, the ventilation valves remained open and the hydrogen concentration was monitored daily.

Daily water samples were taken for ICP-AES analyses throughout the ICET test series. Results from those analyses are presented next. Details of the sample preparation process, typical measurement uncertainties, and minimum detection limits are given in Appendix A. Aluminum was one of the elements measured in the test solution. Tests #1 and #5 were the only experiments that produced a detectable amount of aluminum in solution (Figure 4-10). Both tests were in a pH range of high aluminum solubility and both experienced aluminum coupon corrosion. Test #1 had a higher pH which allows for greater corrosion and consequent solubility

than that of Test #5. Although Test #4 also possessed conditions favorable for aluminum corrosion and solubility, it had different overall solution chemistry because it contained cal-sil as 80% of the insulation source. This difference in chemical composition for Test #4 provided a “passivation” (see Section 4.4.3) layer that protected the aluminum coupons from significant corrosion and resulted in lower aluminum concentrations.

Unfiltered Aluminum Concentration

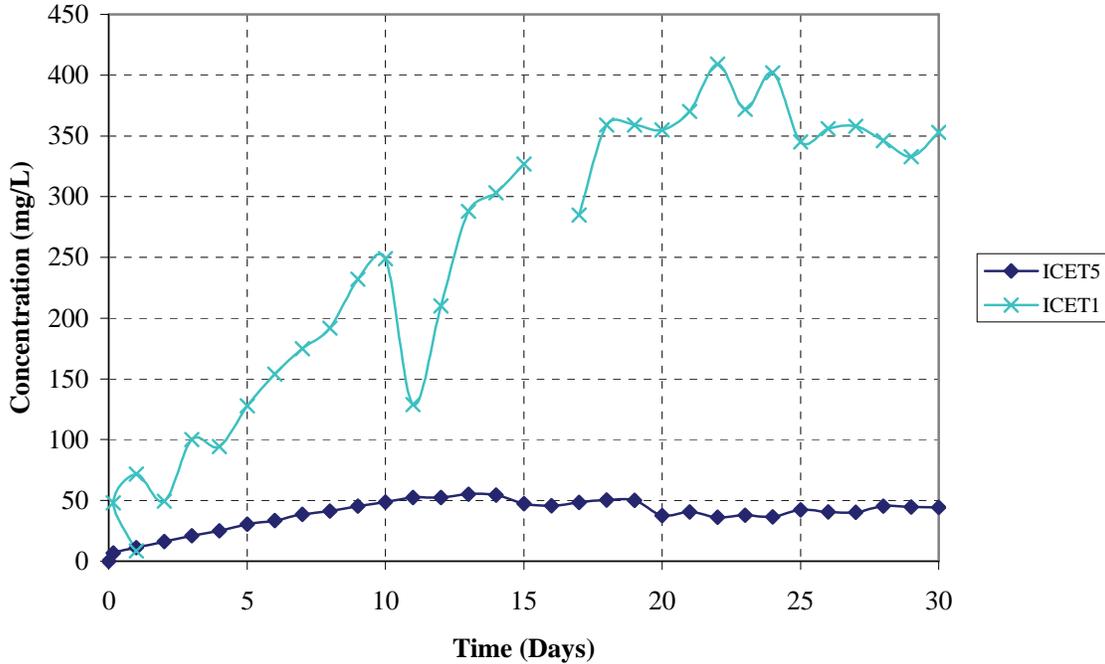


Figure 4-10. ICET Tests #1 and #5 unfiltered aluminum concentration.

As seen in Figure 4-11, Tests #3 and #4 calcium concentrations were the highest of the ICET tests, which is a result of the cal-sil added to solution. Although the amount of cal-sil added was the same for Tests #3 and #4, the systems differed in their initial chemical compositions.

Unfiltered Calcium Concentration

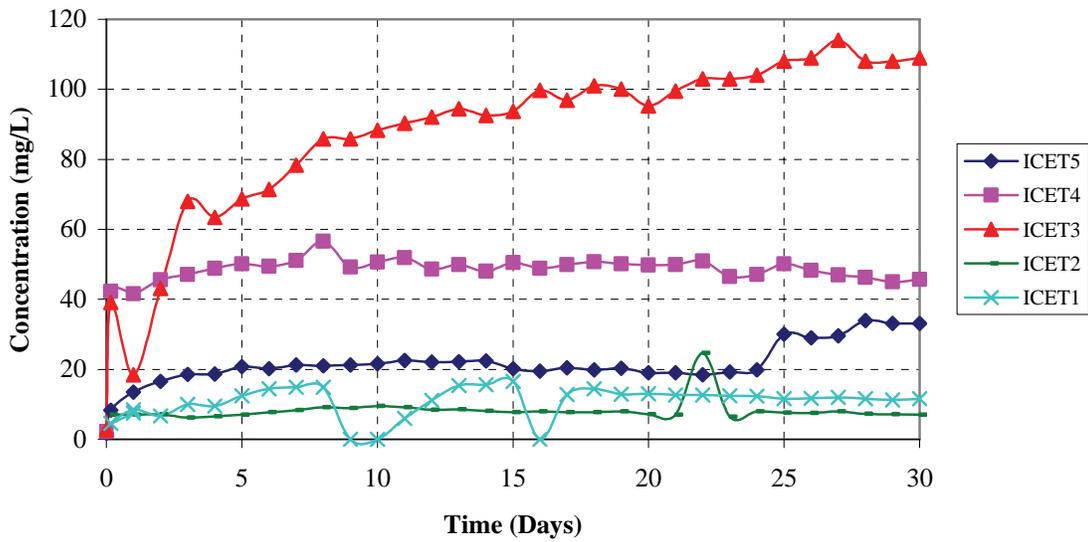


Figure 4-11. ICET unfiltered calcium concentration.

The copper, zinc, and magnesium concentrations, Figures 4-12 through 4-14, were present in low amounts which was not unexpected.

Unfiltered Copper Concentration

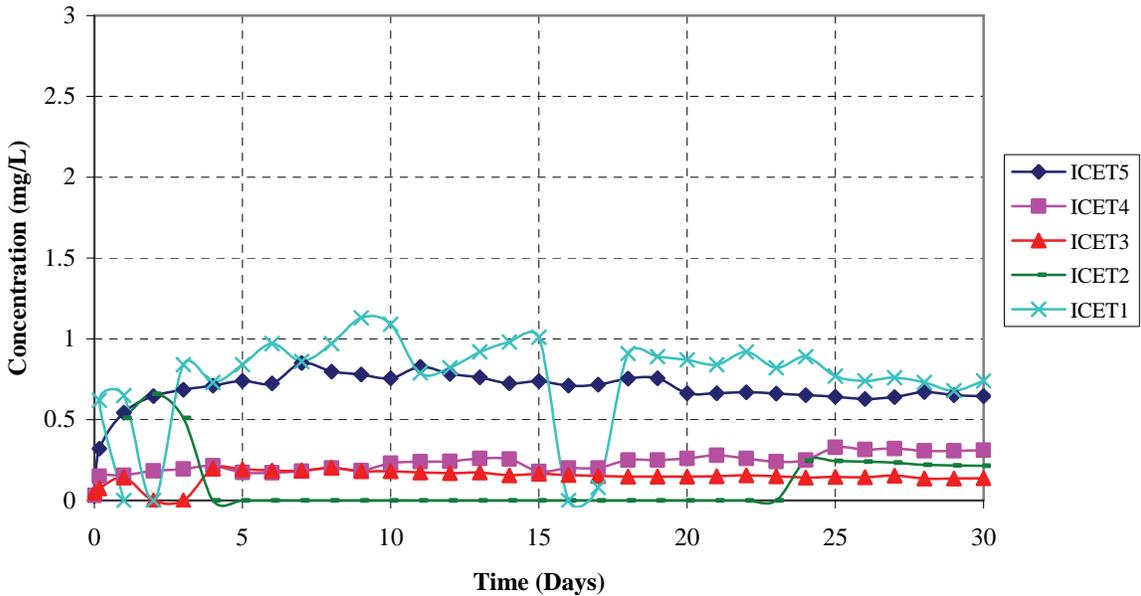


Figure 4-12. ICET unfiltered copper concentration.

Figure 4-13 shows that the highest zinc concentration (up to 10 mg/L) occurred during the first 4 days of Test #2, and then decreased to below 1 mg/L for the remainder of the test. This result was likely caused by a combination of passivation of the zinc surface and settling/precipitation of zinc products. The precipitation was likely caused by phosphate, as the sediment sample from Test #2 contained zinc and phosphate compounds (Figure 4-82).

Unfiltered Zinc Concentration

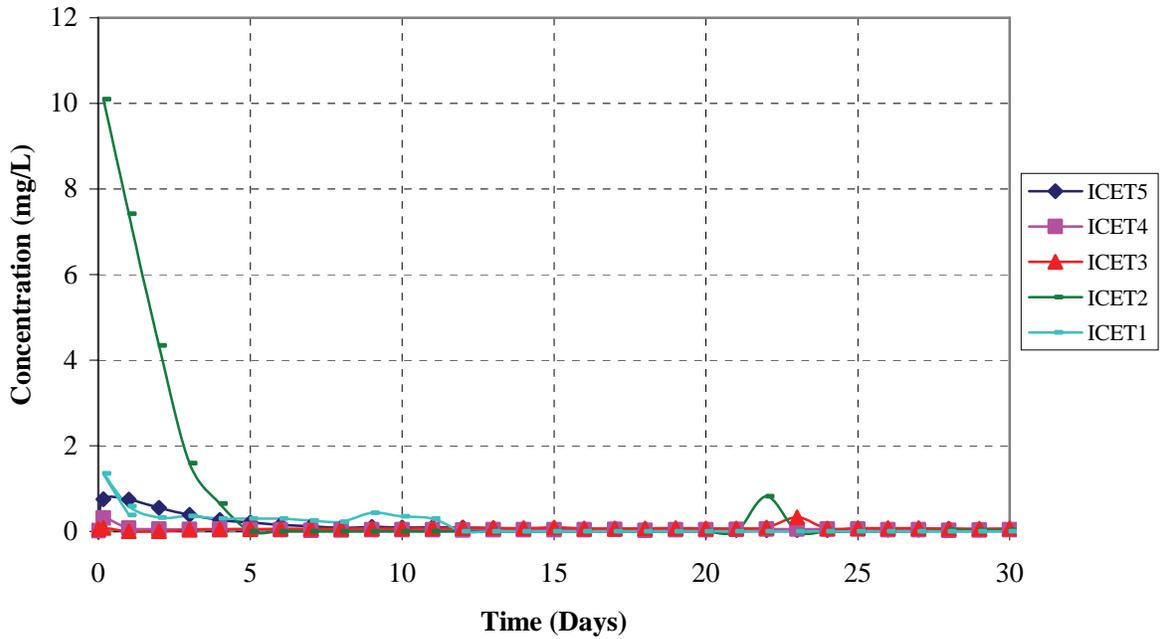


Figure 4-13. ICET unfiltered zinc concentration.

The magnesium concentrations for Tests #2, #3, and #5 are shown in Figure 4-14. The concentrations in Tests #1 and #4 were below minimum detection limits.

Unfiltered Magnesium Concentration

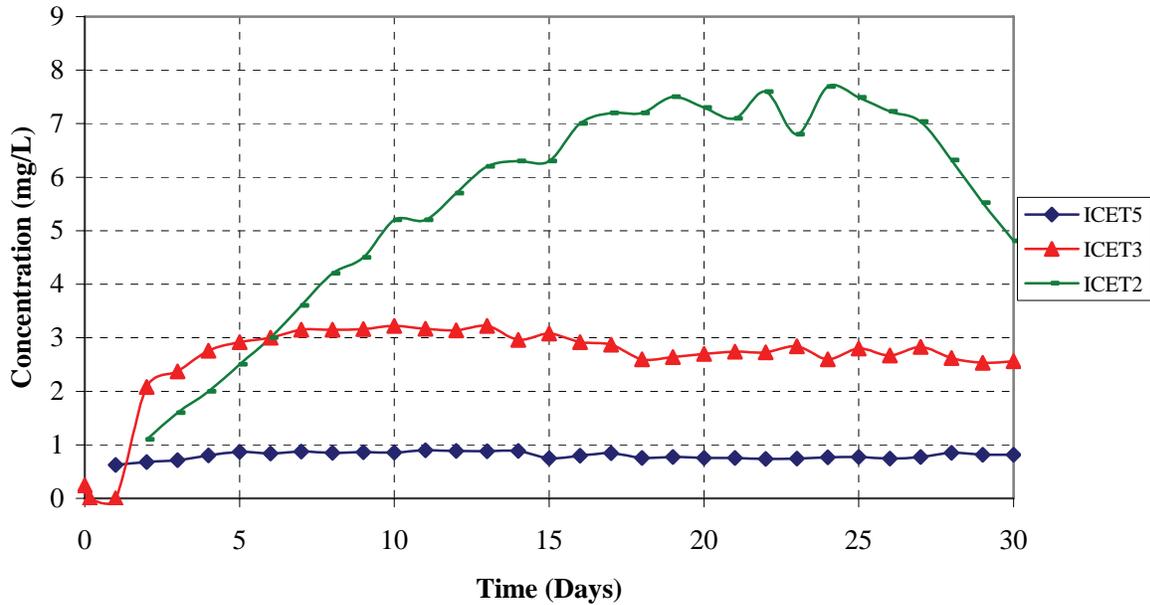


Figure 4-14. ICET Tests #2, #3, and #5 unfiltered magnesium concentration.

As shown in Figure 4-15, Tests #3 and #4 had the largest soluble silica concentrations, which is a result of the presence of cal-sil in the tests. As pH increases, silica solubility increases. Since Test #4 was conducted at a high pH and had cal-sil in solution, it had the highest silica concentrations. Test #2 silica concentrations could be attributed to the dissolution of fiberglass in solution at the system pH. Although Tests #2 and #3 had different sources of insulation, they share similar solution pH and have similar silica concentrations at the end of the tests.

Unfiltered Silica Concentration

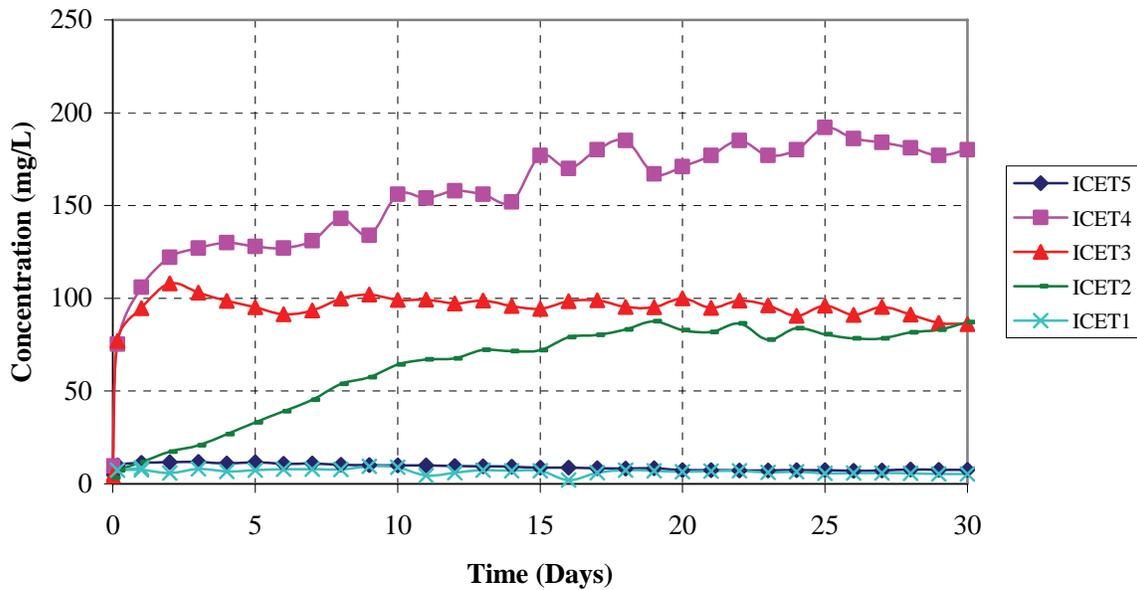


Figure 4-15. ICET unfiltered silica concentration.

Sodium concentrations are presented in Figure 4-16. In Tests #1 and #4, higher concentrations of sodium were detected because sodium hydroxide was added in those tests. A higher concentration of sodium hydroxide was used in Test #4 to obtain a higher pH value than Test #1. An unusually high concentration of sodium was present in Test #4, however, of which only about half can be attributed to the sodium hydroxide addition. The additional sodium could be a result of degrading insulation. At higher pH values as that of Test #4, silica is more soluble (Figure 4-15) which can allow for the insulation, to degraded to a greater degree releasing chemicals such as sodium, into solution.

Tests #1 and #4 had similar system pH values and contained similar constituents. However, the unusually large sodium concentration in Test #4 was not observed in Test #1. It is plausible that the difference in initial silica concentration affected the overall solution chemistry. Test #1 had a smaller amount of initial soluble silica and notable aluminum corrosion. The aluminum could have coated the fiberglass, interacting with the silica content, protecting it from degradation and the subsequent release of silica and sodium into solution. In Test #4, the initial soluble silica resulting from the addition of cal-sil, may have protected the aluminum from corrosion. This phenomenon is discussed in Section 4.4.3. Without corroded aluminum to protect the insulation by interacting with the silica content, the insulation may have been able to dissolve into solution releasing more sodium and silica into solution.

Unfiltered Sodium Concentration

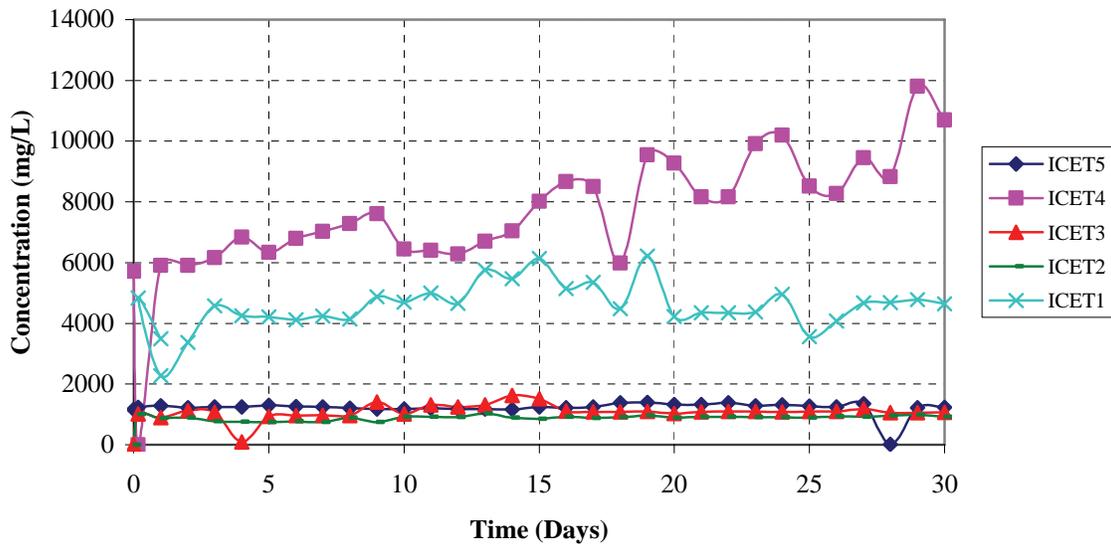


Figure 4-16. ICET unfiltered sodium concentration.

4.1.2. Precipitates

White precipitates were observed in Tests #1 and #5 test solution samples as the solutions cooled to room temperature. No precipitates were observed at 60°C. In Test #1, visual precipitates formed early in the test upon cooling and the quantity increased over the course of the test. In Test #5, the precipitates took longer to form (several days), and the quantities were much smaller than in Test #1. In addition, the quantity of Test #5 precipitates was relatively unchanged throughout the test. ICP-AES analyses were performed to characterize the precipitates from both tests. Because of the larger precipitate formation observed in Test #1, additional analyses were performed in an effort to characterize the precipitate.

ICP-AES analysis was performed to determine the elemental compositions of the precipitates observed in Test #1. Samples from Day 7 and Day 29 were analyzed (Table 4-2). The precipitates were isolated by centrifuge and then thoroughly rinsed with RO water. The ICP-AES results indicated that the precipitate was composed largely of aluminum and boron. Calcium and sodium were also present in smaller amounts. Note that ICP analysis cannot detect the presence of either oxygen or hydrogen in materials. Laboratory tests were performed to produce a surrogate precipitate by titrating aluminum nitrate solution into a pH-9.5 solution containing 2800 mg/L boron and sodium hydroxide at 25°C. Nuclear magnetic resonance analysis was performed on this surrogate precipitate, which indicated that a complexation between aluminum and boron occurred when the solution cooled below 40°C.

Table 4-2. Composition of Precipitates from Test #1

Element	11/28 (Day 7) Precipitate (mg/kg)	12/20 (Day 29) Precipitate (mg/kg)
Al	219,000	220,000
B	84,200	245,000
Ca	23,300	8340
Cu	755	232
Mg	N/D ^a	N/D ^a
Silica	1740	7360
Zn	373	41
Na	23,200	55,300

^aN/D = Not detected

Test #5 precipitates from Days 7 and 29 were also collected and taken for ICP-AES analysis. As shown in Table 4-3, the precipitate was largely composed of aluminum and boron, with smaller amounts of sodium and calcium.

Table 4-3. Composition of Precipitates from Test #5

Element	08/02 (Day 7) Precipitate (mg/kg)	08/24 (Day 29) Precipitate (mg/kg)
Al	143,000	122,000
B	119,000	125,000
Ca	43,200	42,000
Cu	1,790	944
Mg	N/D ^a	712
Silica	2,120	780
Zn	551	112
Na	25,200	20,500

^aN/D = Not detected

When comparing the precipitates from Tests #1 and #5 (Table 4-4), it is seen that both have similar chemical compositions. Test #1 precipitate had a larger concentration of aluminum than in Test #5. Test #5 precipitate had a larger concentration of calcium. These results are supported by the relative amounts of aluminum and calcium in solution (Figures 4-10 and 4-11).

Table 4-4. Distribution of Main Elemental Components of Precipitates from Tests #1 and #5

Precipitate Sample	Al (%)	B (%)	Ca (%)	Na (%)	Other
Test #1					
11/28 (Day 7)	62	24	7	7	0
12/20 (Day 29)	41	46	2	10	1
Average	51	35	5	8	0.5
Test #5					
08/02 (Day 7)	43	36	13	8	0
08/24 (Day 29)	39	40	13	7	0
Average	41	38	13	8	0